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UTILITY
PATENT APPLICATION
TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No.	Beiersdorf 516	Total Pages
First Named Inventor or Application Identifier		
Bernhard MUSSIG		
Express Mail Label No.	EL106680468US	

A
JUL 18 1998
U.S. PTO
156886

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

1. Fee Transmittal Form
(Submit an original, and a duplicate for fee processing)
2. Specification [Total Pages 22]
(preferred arrangement set forth below)
 - Descriptive title of the Invention
 - Cross References to Related Applications
 - Statement Regarding Fed sponsored R & D
 - Reference to Microfiche Appendix
 - Background of the Invention
 - Brief Summary of the Invention
 - Brief Description of the Drawings (if filed)
 - Detailed Description
 - Claim(s)
 - Abstract of the Disclosure
3. Drawing(s) (35 USC 113) [Total Sheets 1]
4. Oath or Declaration [Total Pages 2]
 - a. Newly executed (original or copy)
 - b. Copy from a prior application (37 CFR 1.63(d))
(for continuation/divisional with Box 17 completed)
(Note Box 5 below)
 - i. DELETION OF INVENTOR(S)
Signed statement attached deleting
inventor(s) named in the prior application,
see 37 CFR 1.63(d)(2) and 1.33(b).
5. Incorporation By Reference (useable if Box 4b is checked)
The entire disclosure of the prior application, from which a
copy of the oath or declaration is supplied under Box 4b,
is considered as being part of the disclosure of the
accompanying application and is hereby incorporated by
reference therein.

ADDRESS TO: Assistant Commissioner for Patents
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Washington, DC 20231

6. Microfiche Computer Program (Appendix)
7. Nucleotide and/or Amino Acid Sequence Submission
(if applicable, all necessary)
 - a. Computer Readable Copy
 - b. Paper Copy (identical to computer copy)
 - c. Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

8. Assignment Papers (cover sheet & document(s))
9. 37 CFR 3.73(b) Statement Power of Attorney
(when there is an assignee)
10. English Translation Document (if applicable)
11. Information Disclosure Statement (IDS)/PTO-1449 Copies of IDS
Citations
12. Preliminary Amendment
13. Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
14. Small Entity Statement filed in prior application,
Statement(s) Status still proper and desired
15. Certified Copy of Priority Document(s)
(if foreign priority is claimed)
16. Other:

17. If a CONTINUING APPLICATION, check appropriate box and supply the requisite information:

 Continuation Divisional Continuation-in-part (CIP) of prior application No: _____

18. CORRESPONDENCE ADDRESS

 Customer Number or Bar Code Label  or Correspondence address below

(Insert Customer No. or Attach Bar Code Label here)

NAME	Kurt G. Briscoe, Reg. No. 33,141 SPRUNG KRAMER SCHAEFER & BRISCOE				
ADDRESS	660 WHITE PLAINS ROAD				
CITY	TARRYTOWN	STATE	NY	ZIP CODE	10591
COUNTRY	USA	TELEPHONE	(914)332-1700		FAX (914) 332-1844

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Form 1-49 U.S.P.T.O. 09/15/98UTILITY
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FEE TRANSMITTAL

Note Effective October 1, 1997
Patent fees are subject to annual revision

TOTAL AMOUNT OF PAYMENT (\$ 790.00)

Complete If Known

Application Number	
Filing Date	HEREWITH
First Named Inventor	Bernhard MUSSIG
Group Art Unit	
Examiner Name	
Attorney Docket Number	Beiersdorf 516-KGB

METHOD OF PAYMENT (check one)

1. The Commissioner is hereby authorized to charge indicated fees and credit any over payments to
 Deposit Account Number 19-3869
 Deposit Account Name SPRUNG KRAMER SCHAEFER & BRISCOE
 Charge Any Additional Fee Required Under 37 CFR 1.16 and 1.17 Charge the Issue Fee Set in 37 CFR 1.18 at the Mailing of the Notice of Allowance

2. Payment Enclosed.
 Check Money Order Other

FEE CALCULATION

1. FILING FEE

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description	Fee Paid
101	790	201 395 Utility filing fee	790.
106	330	206 165 Design filing fee	
107	540	207 270 Plant filing fee	
108	790	208 395 Reissue filing fee	
114	150	214 75 Provisional filing fee	
SUBTOTAL (1) (\$)			790.

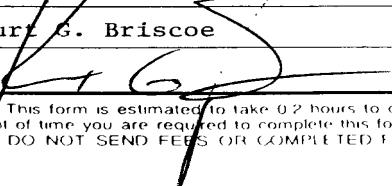
Total Claims	Extra	Fee from below	Fee Paid
19	-20	= 0	X =
Independent 1	- 3	= 0	X =
Claims			
Multiple Dependent Claims		X	=

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description
103	22	203 11 Claims in excess of 20
102	82	202 41 Independent claims in excess of 3
104	270	204 135 Multiple dependent claim
109	82	209 41 Reissue independent claims over original patent
110	22	210 11 Reissue claims in excess of 20 and over original patent
SUBTOTAL (2) (\$)		

3. ADDITIONAL FEES

Large Entity Fee Code (\$)	Small Entity Fee Code (\$)	Fee Description	Fee Paid
105	130	205 65 Surcharge - late filing fee or oath	
127	50	227 25 Surcharge - late provisional filing fee or cover sheet	
139	130	139 130 Non-English specification	
147	2,520	147 2,520 For filing a request for reexamination	
112	920*	112 920* Requesting publication of SIR prior to Examiner action	
113	1,840*	113 1,840* Requesting publication of SIR after Examiner action	
115	110	215 55 Extension for reply within first month	
116	400	216 200 Extension for reply within second month	
117	950	217 475 Extension for reply within third month	
118	1,510	218 755 Extension for reply within fourth month	
128	2,060	228 1,030 Extension for reply within fifth month	
119	310	219 155 Notice of Appeal	
120	310	220 155 Filing a brief in support of an appeal	
121	270	221 135 Request for oral hearing	
138	1,510	138 1,510 Petition to institute a public use proceeding	
140	110	240 55 Petition to revive - unavoidable	
141	1,320	241 660 Petition to revive - unintentional	
142	1,320	242 660 Utility issue fee (or reissue)	
143	450	243 225 Design issue fee	
144	670	244 335 Plant issue fee	
122	130	122 130 Petitions to the Commissioner	
123	50	123 50 Petitions related to provisional applications	
126	240	126 240 Submission of Information Disclosure Stmt	
581	40	581 40 Recording each patent assignment per property (times number of properties)	
146	790	246 395 Filing a submission after final rejection (37 CFR 1.129(a))	
149	790	249 395 For each additional invention to be examined (37 CFR 1.129(b))	
Other fee (specify) _____			
Other fee (specify) _____			
Reduced by Basic Filing Fee Paid			SUBTOTAL (3) (\$ 790.00)

SUBMITTED BY

Typed or Printed Name	Kurt G. Briscoe
Signature	

Complete (if applicable)

Reg Number	33,141
Deposit Account User ID	19-3869

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Beiersdorf 516-KGB:lad
1120-Dr.Di-hf

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants : Bernhard MÜSSIG
Serial No. :
Filed : Herewith
For : SELF-ADHESIVE PROTECTIVE FILM WITH OLEFIN
RUBBER ADHESIVE
Group Art Unit :
Examiner :

September 18, 1998

Hon. Commissioner for Patents
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Prior to examination, kindly amend the above-identified application as follows:

IN THE CLAIMS

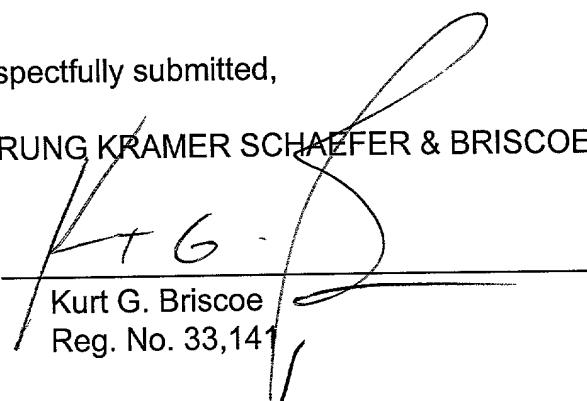
Claim 18, line 1, cancel "Claims 1 to 17" and substitute --Claim 1--.

REMARKS

The foregoing amendment serves to eliminate multiple dependencies.

Respectfully submitted,

SPRUNG KRAMER SCHAEFER & BRISCOE

By: 

Kurt G. Briscoe
Reg. No. 33,141

660 White Plains Road
Tarrytown, NY 10591-5144
(914) 332-1700

BEIERSDORF AG
Hamburg

5

Description

“Self-adhesive protective film with olefin rubber adhesive”

10 The invention relates to a self-adhesive protective film for protecting the fresh paint finish of vehicles such as cars and for protecting freshly painted vehicle components against soiling and damage during assembly, transportation and storage, and to its use on vehicles, especially on cars.

15 The preservation and protection of motor vehicles in the course of transportation from manufacturer to dealer has long been common practice.

20 It is known that, in order to preserve cars, paraffin waxes are applied in a thickness of from 5 to 20 µm. However, it has been found that, especially on horizontal areas of the vehicles, a thin and usually non-uniform layer of this nature does not afford adequate protection against external influences such as, for example, the corrosive action of bird droppings.

25 A considerable disadvantage of sealing with paraffin wax is the need to remove the preservative using a steam jet, surfactants or solvents. Environmentally sound recovery and disposal of the residues are the cause of great complexity in terms of apparatus, and of very high costs to boot.

30 Self-adhesive surface protective films for motor vehicles have also been known for some time. In the majority of cases these are permanently adhesive films for protection and decoration, such as an anti-stonechip film, for example. These self-adhesive films remain permanently on the vehicle following their application.

35 Reversibly adhering self-adhesive films for protecting the car during transportation have so far been used only in a few isolated cases and without great success. The object of the invention was to remedy this situation and, in particular, to provide a self-adhesive protective film which does not have the disadvantages of the prior art, or at least not to the same extent. This object is achieved by a self-adhesive protective film as 40 characterized in more detail in the patent claims.

Description of the invention

The adhesive film of the invention is a protective film for use on painted vehicles, especially cars, in particular having an adhesive comprising an essentially amorphous copolymer of at least two different α -olefin monomers with a number of in each case from 2 to 12 carbon atoms and at least one diene as comonomer, where no α -olefin should have a proportion of 75 mol% or more, and having a bond strength of from 0.2 to 5 2.8 N/cm, preferably from 0.3 to 1.5 N/cm on steel (AFERA Method 4001, corresponding to DIN EN 1939). The number of carbon atoms of the α -olefins is between 2 and 12. Examples are linear alkenes, such as ethylene, propene, 1-butene, 1-hexene and 1-octene, and isoalkenes, such as 3-methyl-1-butene or 4-methyl-1-pentene. Suitable dienes are those 10 common for the preparation of EPDM rubber, examples being 1,4-hexadiene, dicyclopentadiene or, in particular, 5-ethylidene-2-norbornene (ENB). The proportion of diene in the polymer is preferably between 0.5 15 and 10% by weight. The proportion of the further comonomers (third and any other comonomers) should in particular be below 30 mol% if they are 20 non-olefinic comonomers. The latter comonomers may be polar, such as vinyl acetate, esters of acrylic or methacrylic acid, vinyl chloride, vinyl ethers or acrylonitrile, or apolar, such as isobutene, styrene or α -olefin.

The Mooney viscosity ML (1+4) 125°C should be below 50, preferably 25 below 30, so that the viscosity of the melt or solution is not too high. It is tested in accordance with ASTM D 1646.

It is advantageous to crosslink the adhesive composition. This increases the thermal stability (under alternating climatic conditions, for example); 30 peel increase on rough substrates or apolar substrates, such as olefin rubber window seals, is reduced. For crosslinking, suitable crosslinking agents are those customary for EPDM rubber. Preference is given to the technique of radiation crosslinking, especially with UV radiation and electron beams. In the case of UV radiation, the adhesive composition is 35 irradiated from the adhesive side. For the use of photoinitiators or the addition of crosslinking promoters such as esters of allyl alcohol, of methacrylic or of acrylic acid to the adhesive composition it is possible to reduce the radiation dose.

To high a proportion of polar non-olefinic monomers, however, increases the sticking force to the painted metal vehicle panel following storage under alternating climatic conditions. It is therefore advantageous to keep the

5 proportion of polar comonomers below 20 mol% and, in particular, to omit them entirely; in this case, then, the copolymer comprises at least three apolar monomers.

The proportion of each of the α -olefinic monomers should be below

10 75 mol%; more favourable is a proportion of not more than 65 mol%. The most suitable are terpolymers comprising three α -olefins, whose proportion of α -olefinic monomers is in each case between 5 and 60 mol%. Limiting the proportion of individual monomers avoids increased sticking forces following storage under alternating climatic conditions, avoids poor tack on

15 application, and avoids deformation of the paintwork. Deformations comprise visually perceptible and irreversible changes to the painted surface, which result when the fresh, not yet fully cured paint has been covered with an inappropriate protective film.

20 In order to optimize its properties, especially its adhesion to specific paints, the self-adhesive composition employed can be blended with one or more additives, such as tackifiers (resins), plasticizers, fillers, pigments, UV absorbers, light stabilizers, anti-ageing agents, crosslinking agents, crosslinking promoters or elastomers.

25 Examples of such elastomers are EPM rubber, polyisobutylene, butyl rubber, hydrogenated block copolymers of styrene and dienes, or acrylate copolymers. Examples of suitable tackifiers are hydrocarbon resins (comprising unsaturated C5 or C7 monomers, for example), terpene-phenolic resins, terpene resins made from raw materials such as α - or β -pinene, aromatic resins, such as indene-coumarone resins, or resins of styrene or α -methylstyrene, such as rosin and its derivatives, such as disproportionated, dimerized or esterified resins, it being possible to employ glycols, glycerol or pentaerythritol, and also others as listed in

30 35 Ullmanns Enzyklopädie der technischen Chemie, Volume 12, pp. 525-555 (4th ed.), Weinheim. Resins which are stable to ageing and have no olefinic double bond are particularly suitable, examples being hydrogenated resins.

Suitable fillers and pigments are, for example, carbon black, titanium dioxide, calcium carbonate, zinc carbonate, zinc oxide, silicates or silica.

5 Suitable UV absorbers, light stabilizers and anti-ageing agents for the adhesive compositions are the same as those listed below for the stabilization of the film.

10 Examples of suitable plasticizers are aliphatic, cycloaliphatic and aromatic mineral oils, diesters or polyesters of phthalic acid, trimellitic acid or adipic acid, liquid rubbers (e.g. nitrile rubbers or polyisoprene rubbers), liquid polymers of butene and/or isobutene, acrylates, polyvinyl ethers, liquid resins and soft resins based on the raw materials for tackifier resins, wool wax and other waxes, or liquid silicones. Plasticizers which are stable to 15 ageing and have no olefinic double bond are particularly suitable.

20 Crosslinking agents are, for example, phenolic resins or halogenated phenolic resins, melamine resins and formaldehyde resins. Examples of suitable crosslinking promoters are maleimides, allyl esters, such as triallyl cyanurate, and polyfunctional esters of acrylic and methacrylic acid.

25 For some paint systems it is advantageous to add constituents of the paints to the adhesive in order to reduce the migration of paint constituents (such as fatty acid esters) from the paint into the adhesive composition. Paint constituents of this kind are described in more detail in EP 0 763 584 and US 5,612,135.

It is advantageous, moreover, for recycling and energy recovery if the adhesive and, in particular, the entire protective film is free from halogen.

30 The backing used for the adhesive comprises thermoplastic films, preferably polyolefin films. These may consist, for example, of polyethylene, polypropylene and their mixtures or copolymers (e.g. random copolymers). Appropriate thicknesses for the backing films are from 20 to 35 80 µm (including, if appropriate, the adhesion promoter layer). The softness of the backing film is relevant to the flexibility of the protective film during application; the force at 10% extension should not exceed 25 N/15 mm, preferably 16 N/15 mm, either in the lengthwise or transverse

direction. On account of their stability to shrinkage under hot conditions and their good flexibility, propylene copolymers are particularly suitable. For improving the adhesion between film and adhesive it is advantageous, although not absolutely necessary, to use an adhesion promoter layer; in 5 that case the backing film consists of the base layer and the adhesion promoter layer. The latter consists preferably of polymers formed from predominantly olefinic monomers and/or components of base layer and/or adhesive. With particular preference the adhesion promoter comprises at 10 least one polymer of which at least 50 mol% consists of one or more α -olefins. Examples of such polymers are thermoplastic EPM or EPDM elastomers, ionomers based on ethylene-(meth)acrylic acid copolymer, or maleic anhydride-modified polyolefins.

In order to provide the backing film with weathering stability it is sensible - 15 and, in the case of polyolefins, generally vital - to add light stabilizers. In the context of application to the protective film, their function is primarily to avoid embrittlement of the backing film. In the case of a multilayer backing film the protection relates in particular to the base layer. Light stabilizers of 20 this kind are described in Gaechter and Müller, Taschenbuch der Kunststoff-Additive [Pocket Book of Plastics Additives], Munich 1979, in Kirk-Othmer (3rd) 23, 615-627, in Encycl. Polym. Sci. Technol. 14, 125-148 and in Ullmann (4th) 8, 21; 15, 529, 676. Hindered amine light stabilizers (HALS) in particular, such as CAS Nos. 106990-43-6, 65447-77-0, 70624-18-9 or 52829-07-9, are appropriate for the protective film of the 25 invention. The amount of light stabilizer should be at least 0.15, preferably at least 0.30, % by weight, based on the backing film. The use of antioxidants for the film (e.g. Irganox 1010, or trisnonylphenyl phosphite) is advantageous but not absolutely necessary. Further suitable UV absorbers, light stabilizers and anti-ageing agents are listed in 30 EP 0 763 584 on page 5.

The light stabilizers referred to for the film can also be employed for the adhesive composition. Such an addition raises the ageing stability of the adhesive composition, especially under UV exposure of more than 100 kLY 35 (kilolangleys), but is generally unnecessary for applications entailing less than 6 months of outdoor weathering. In the case of the polyisobutylene homopolymer adhesives or butyl rubber adhesives which are not in

accordance with the invention, such an addition is vital to prevent residues of adhesive under very severe UV exposure.

The light stability of backing film and adhesive can also be improved by 5 other additives in the protective film, especially in the base layer of the backing film. Such an improvement can be brought about by UV absorbers (e.g. Tinuvin P, Ciba) or, primarily, by means of reflective pigments (e.g. titanium dioxide). The protective film of the invention is intended as a high-grade branded product and therefore has a permeability for UV radiation in 10 the range from 290 to 360 nm of less than 1%, preferably of less than 0.1%.

15 The adhesion promoter can be applied by coextrusion with the base film or by coextrusion with base film and adhesive, or by coating onto the base film.

One advantageous embodiment is to apply the adhesion promoter simultaneously with the extrusion of the film, so that the resulting coextruded film need only be coated with adhesive. The optimum technical 20 solution for the protective film of the invention is the simultaneous coextrusion of film and adhesive (including, if appropriate, an adhesion promoter layer).

25 The particular significance of this invention, although residing *inter alia* in the process for preparing the protective film and in the composition of the backing film, consists primarily in the composition of the pressure-sensitive adhesive layer of the invention and in the combination of these factors.

Prior art relating to car protection films

30 When tested by applying them to painted metal panels and storing these panels under alternating climatic conditions, known apolar uncrosslinked self-adhesive compositions show better paint compatibility than polar compositions. By paint compatibility is meant absence of paint film 35 deformation and ease of peel following storage under alternating climatic conditions. On the other hand, however, such compositions are too soft (insufficiently cohesive) and show residues of composition when the film is peeled off. If, therefore, such compositions are crosslinked by chemical or

radiative means, they lead to unwanted paint film deformation. An exception is formed by self-adhesive compositions based on natural rubber resin, which can be peeled off again without residue and with only minor paint film deformation. However, these compositions are not stable to

5 ageing on exposure to UV radiation. This leads to severe, greasy or hard paintlike residues on the coated metal panel following extreme stresses over a prolonged period (from three to six months) as occur, for example, in Florida.

10 Also known are polyacrylate compositions. If uncrosslinked polyacrylate compositions are subjected to storage under alternating climatic conditions they exhibit good compatibility with paint surfaces. On the other hand, however, they are of such little cohesion that when the film is peeled off they leave residues of the adhesive composition on the substrate. If these

15 compositions are strongly crosslinked chemically or by radiation, they can be peeled off without residue but, on the other hand, cause permanent deformations on the paint surface and in some cases, moreover, show inadequate adhesion when bonded to curved painted areas.

20 Japanese Patent Application JP 02 199 184 discloses a radiation-crosslinkable pressure-sensitive adhesive which for protective use on paint films must be formulated with a low glass transition temperature in order to prevent permanent deformations of the paint film. In this case there are problems owing to the inadequate adhesiveness of the self-adhesive film

25 on the vehicles during application and transportation.

This also applies to acrylates as described in US 5,612,136. The process of irradiation disclosed therein, moreover, damages the film, thus reducing its weathering stability, if irradiation is carried out for so long that the

30 residual monomers are fully removed by polymerization. At lower doses of irradiation, the content of residual monomers is so high that they migrate into the vehicle finish and thereby cause it to swell. A further disadvantage is the migration of photoinitiators into the paint, where they reduce its UV stability.

35 European Patent Application EP 0 519 278 discloses, for protecting cars, a film comprising a backing which is coated with a rubber-based pressure-sensitive adhesive, based especially on polyisobutylene, having a dynamic

modulus of elasticity of from 2×10^5 to 7×10^6 dyn/cm², corresponding in SI units to a value of from 2×10^4 to 70×10^4 Pa, at 60°C. Performance tests conducted with a self-adhesive film of this kind indicate relatively good compatibility with paint surfaces. The adhesiveness following storage

5 under alternating climatic conditions on paints common in the car industry, however, is so low that the bond strength required in practice is not always adequately attained. Especially under the influence of moisture on the film, the bond strength is frequently reduced to such an extent that the film becomes detached, during transportation, from the vehicle it is protecting,

10 so that there is no longer any protective effect. Furthermore, the self-adhesive composition shows no compatibility with the sealing rubbers (sealing profiles) which are common in carmaking; when the protective film is peeled away from window profiles, residues of the adhesive coating composition remain on the rubber. When the protective film is peeled off,

15 edges with residues of adhesive remain, which can be traced back to the breakdown of the adhesive under UV weathering.

JP 95-325285 describes a similar product. Polyisobutylene is again proposed as the adhesive composition, although it may include butyl rubber (polyisobutylene with double bonds).

European Patent Application EP 0 592 913 describes a surface protection film for the transportation of cars, which is composed of a copolymer of unsaturated, polar comonomers and α -olefins, especially of EVAc. In the

25 case of this protective film there is no application of an additional adhesive coating. A film of this kind, while admittedly of simple construction, nevertheless displays distinct disadvantages in terms of its use. The bond strength is very low; this defect can be remedied by heating in the course of application. In addition, unless these films are provided with a protective

30 layer or a release covering, they lead to blocking, as taught by EP 0 768 356. A particularly serious consideration is that the films described show very severe paint film deformation and very great difficulty of removal following use.

35 The object, therefore, is to find an adhesive film which possesses sufficiently high initial bond strength that it can be bonded under tension (application of the adhesive film to curved areas) without becoming detached and yet following prolonged storage (up to one year under

weathering) can still easily be peeled off again. Accordingly, there has been no lack of attempts to remedy this deficiency. The EVAc adhesive film described in DE 195 32 220 is markedly superior to the product described in EP 0 592 913, but when peeled off after use still shows a 5 bond strength which is too high to allow the adhesive film to be peeled off without tearing and without excessive effort by the user. With one-component paints, moreover, there is paint film deformation.

The increase in bond strength following outdoor storage, which the skilled 10 worker calls peel increase, is caused by interactions - especially polar forces - of the adhesive with the paint. The skilled worker will therefore avoid polar comonomers such as acrylate or vinyl acetate. The polyisobutylene referred to in the abovementioned EP 0 519 278 is an adhesive which is appropriate in this context. The disadvantage, however, 15 is the ageing behaviour. Polyisobutylenes are known to be soft (of low cohesion), especially following ageing as induced by heat or UV radiation. This leads to residues on the paint following removal. For this reason, in the case of the commercial adhesive film based on polyisobutylene, the films are highly pigmented - the destructive influence of light should be 20 kept away from the adhesive as far as possible. In the edge region, however, the adhesive is exposed directly to UV light, leading to severe residues there. Attempts to avoid this are made by adding antioxidants and hindered amine light stabilizers (HALS) to the polyisobutylene; these additives are described in US 5,601 917.

25 Adhesives comprising hydrogenated styrene-diene block copolymers are substantially more stable to UV, and their use is described in the utility model DE 296 04 473 U1. A significant disadvantage of such block copolymers is their thermally reversible crosslinking via the styrene 30 domains. If an adhesive film prepared from these polymers is peeled off in the summer from a vehicle which has become warm in the sun, the adhesive remains to a large extent adhering to the paintwork, since the cohesion of the adhesive compositions is less than that of the adhesion to the paintwork. Heating leads to shrinkage of the protective film and, as a 35 result, the adhesive - which is soft under hot conditions - transfers smearingly onto the metal panel at the retreating edges of the adhesive film.

EP 0 661 364 describes a surface protection film comprising an adhesive composition which exhibits "strong initial tack" (p. 2, line 6) and is therefore inappropriate for the purposes of the present invention; pages 3 and 6 describe bond strengths to steel of up to 1 kg/25 mm, corresponding

5 approximately to AFERA sticking forces to steel of up to 4.0 N/cm. To limit the peel increase behaviour on exterior weathering and/or storage under alternating climatic conditions it is important to restrict the proportion of polar comonomers as a whole and to limit the proportion of each individual α -olefin monomer, something which is not undertaken by the cited

10 invention. The films described, moreover, are not light stable, since no measures are taken - such as the addition of HALS or light-reflective pigments - to achieve the light stabilization necessary for this utility. The use of protective film on the outside of vehicles exerts particularly stringent requirements in terms of light stability, especially in the UV range, so that

15 for this reason too protective films of this kind are completely unsuited to this utility. On application to vehicles an important part is played, owing to curved areas, by the extensibility of the film: the cited invention, however, emphasizes the suitability for planar areas ("plates" and "boards") and therefore does not attempt to limit the force at 10% extension.

20 WO 96/37568 describes the use of polyhexene and/or polyoctene for an apolar pressure-sensitive adhesive. The polymers described in the examples exhibit little peel increase but owing to their low cohesion, caused by the low molecular weight of customary commercial polymers of

25 this kind, these polymers likewise lead to residues, which it is attempted to avoid by adding other polymers, referred to therein as cold flow restricting agents. For practical purposes, nevertheless, these adhesives are of inadequate cohesion (are too soft), which leads to residues after weathering, especially if the adhesive tape shrinks on exposure to heat.

30 Properties of the protective film of the invention

For the skilled worker it was surprising and unforeseeable that an adhesive composition which consists essentially of a copolymer of at least three

35 monomers, including at least two α -olefins having in each case from 2 to 12 carbon atoms and a proportion of in each case less than 75 mol%, and at least one diene, would have the desired combination of properties:

- low peel increase
- good UV resistance
- no paint film deformation
- sufficient bond strength on application to curved surfaces
- 5 - high cohesion (no residues on peeling or on shrinkage of the protective film).

The very high weathering stability (resistance to UV light), in particular, is a completely surprising property.

10 On the one hand, these self-adhesive compositions feature good strength of adhesion to a variety of coating materials common in the automotive industry, even under the effect of moisture or humidity, with the result that the protective film does not become detached from the vehicle even under
15 the effect of wind or under tension due to bonding to curved surfaces. Furthermore, the self-adhesive composition exhibits a sufficient bond strength within the first few minutes following application, so that after just half an hour, for example, the protective film can be exposed to a fast airstream (up to 160 km/h) and yet, on the other hand, is not difficult to
20 remove following prolonged use. In addition, the self-adhesive composition of the invention ensures a very low unrolling force. In particular, on a 65 µm thick film consisting predominantly of EP copolymer, the self-adhesive composition of the invention shows no paint film deformation on metal panels which have been coated with polyurethane and heat-treated at
25 130°C for 30 minutes and following storage under alternating climatic conditions as detailed in Example 1 of this description.

The peel force of the protective film of the invention from two-component PU coating materials is greater than 0.1 N/cm when fresh and below
30 3 N/cm following storage under alternating climatic conditions (in accordance with AFERA Method 4001). In addition, irradiation of the protective film with UV light, for example with Xenotest 1200 at 55°C for 3000 hours, leads to no impairment to the properties of this protective film. There is no embrittlement of the film, and neither does the self-adhesive
35 composition cause paint film deformation or discoloration and nor do residues of composition occur when the protective film is removed.

The protective film of the invention is therefore particularly suitable for protecting the fresh paint finish of cars during assembly or transportation, or as a protection for freshly painted steel panels during machining and transportation. In this context the protective film can be bonded just half an

5 hour after the coated surfaces have passed through the oven, without any disadvantages whatsoever, despite the fact that at this point in time the paint has still not yet fully cured. In addition, the protective film exhibits outstanding properties when used as edge-fastening tape for the additional fastening of self-adhesive masking films of large surface area.

10 A further feature of the protective film of the invention is that it can be applied in a large width over the bonnet, roof and boot of cars and, owing to its flexibility, conforms very well to planar and even gently curved surfaces. It is thereby possible to protect the horizontal surfaces which are

15 most at risk from soiling. However, even narrow areas such as, for example, the projection of the door below the windows, or bumpers, can easily be covered over. Protection of the vertical surfaces on the vehicle is particularly appropriate during its assembly.

20 The protective film is resistant to sunlight, moisture, heat and cold, with weathering stability of at least six months. The addition of titanium dioxide and of light stabilizers, in particular, leads to an improvement in the UV resistance of the protective film. Even very high sun levels as are encountered, for example, in Florida, do not cause the protective film to fail

25 or detach, at least within a period of six months.

After application of the protective film and following storage for 14 days at alternating temperatures and under the effect of weathering, the self-adhesive composition does not give rise to any deformation of the

30 underlying paint film. In addition, the strength of the protective film in comparison to preservation with wax ensures flawless protection against soiling, such as bird droppings, and against damage to the entire vehicle by minor mechanical events.

35 Following use, the protective film can be removed without residue and without tearing the backing film, despite the good adhesiveness required. Even on the rubber seals used widely on cars there are no residues of adhesive following its removal. Especially if the protective film is free from

halogen, material recycling thereof or energy recovery therefrom is possible.

In the text which follows, the invention will be illustrated on the basis of
5 examples which are not, however, intended to restrict the invention.

Examples

Example 1

10

A film is manufactured by flat-film extrusion in a width of 1450 mm. It consists of a 50 µm thick base layer and 15 µm thick adhesion promoter layer. The base layer consists of 60 parts by weight of PP homopolymer, 30 parts by weight of LLDPE, 10 parts by weight of titanium dioxide and 0.3 15 part by weight of a HALS (Tinuvin 770). The adhesion promoter layer consists of a compound comprising 30 parts by weight of PP homopolymer, 20 parts by weight of LLDPE and 50% by weight of the polymer described below as adhesive.

20

The film has the following physical properties:

Overall thickness of film with adhesion promoter	65 µm	DIN 53370
Overall weight of film with adhesion promoter	63 g/m ²	DIN 53365
Tensile strength, lengthwise	30 N/mm ²	DIN 53455-7-5
Force at 10% extension, lengthwise	19 N/15 mm	DIN 53455-7-5
Tensile strength, transverse	20 N/mm ²	DIN 53455-7-5
Extension, lengthwise	450%	DIN 53455-7-5
Extension, transverse	450%	DIN 53455-7-5
Impact strength under tension, lengthwise	3000 mJ/mm ²	DIN 53448
Impact strength under tension, transverse	200 mJ/mm ²	DIN 53448

The adhesive prepared is a 10% strength solution of a copolymer consisting of 48% by weight each of propene and ethylene and 4% by weight of 5-ethylidene-2-norbornene, having a Mooney viscosity ML (1+4)

125°C of 28 in toluene, and it is applied to the film with a spreader bar and dried for four minutes at 80°C in a tunnel. The resulting protective film is trimmed at the edge and wound up into rolls 200 m long and 1400 mm wide. The thickness of adhesive is 20 µm. The Mooney viscosity M_L (1+8) 5 at 125°C was measured as 35.

The self-adhesive film produced in this way can be unwound easily and without creases and when used to protect cars can be applied without problems. Owing to the good tack and ease of correction, the bonding 10 processes can be accelerated. After use for up to a six-month period of bonding under outdoor weathering, the self-adhesive film can be peeled off again without defects.

15 The protective film is characterized by the physical properties reproduced in the following table.

Overall thickness of protective film:	80 µm
Peel force from the two-component PU paint after 14 days of alternating climatic conditions (cycle 2 as indicated below) at a peel angle of 180° and a peel rate of 300 mm/min:	1.4 N/cm
Peel force from the reverse side at a peel angle of 180° and a peel rate of 300 mm/min:	0.1 N/cm
Bond strength on steel	0.3 N/cm
Bond strength on paint	0.3 N/cm

The alternating climatic conditions comprise the following cycles:

Cycle 1		Cycle 2	
Duration [h]	Temperature [°C]	Duration [d]	Temperature [°C]
4	80	3	90
4	-30	plus 4 times cycle 1	
16	40 at 100% relative humidity		

Cycle 2 is repeated a total of two times here.

5 The protective film was bonded to freshly painted metal panels (2-component PU paint) and peeled off after a week; no paint film deformation was in evidence. Samples bonded to paint were weathered at 97 kLy; following removal, no residues could be seen on the surface or in the edge region.

10 Example 2

A film was produced by the procedure of Example 1, with the base layer consisting of 80 parts by weight of a PP random copolymer with 5.5% of ethylene (Novolen 3300 MC, BASF), 10 parts by weight of LLDPE, 7 parts 15 by weight of titanium dioxide and 0.45 part by weight of a HALS (Chimassorb 944, Ciba) and the adhesion promoter layer consisting of 20 parts by weight of the PP random copolymer and 80 parts by weight of LLDPE. This film was coated by the procedure of Example 1. The adhesive used is a polymer as in Example 1 but with addition of 5% by weight of 20 RegalrezTM 116 (tackifier, Hercules), 10% by weight Levapren 700 (VA elastomer, Bayer), 0.5% by weight Irgacure 651 (Ciba), 1.5% by weight trimethylopropane triacrylate, 0.2% by weight Irganox 1010 (Ciba) and 0.3% by weight Tinuvin 770 (Ciba). The thickness of adhesive applied is 16 µm. The protective film was crosslinked on the adhesive side under 25 nitrogen using a UV lamp (medium-pressure Hg lamp, 120 W/cm) at a distance of 10 cm and at a speed of 10 m/min.

The film exhibit a force of 14 N/15 mm at 10% extension in the lengthwise direction. The bond strength to steel is 0.9 N/cm, to paint 0.9 N/cm (fresh) 30 and 2.6 N/cm (following storage under alternating climatic conditions). The protective film was bonded to freshly painted metal panels (2-component

PU paint) and peeled off after a week; slight deformations of the paint are discernible in the edge region. Samples bonded to paint were weathered at 97 kLy; following removal, no residues could be seen on the surface or in the edge region.

5

Example 3

The protective film was produced by coextrusion of three layers including adhesive. The base layer consists of the raw materials described in 10 Example 3. The adhesive was a polymer formed from 59% by weight ethylene, 37% by weight propene and 4% by weight 5-ethylidene-2-norbornene (granulated and powdered with amorphous silica), having a Mooney viscosity ML (1+4) 125°C of 22, with addition of 0.2% by weight of Irganox 1010 (Ciba) and 3% by weight Escorez 5000, liquid (plasticizer, 15 Exxon).

The extruder for the base layer was charged with the raw materials set out in Example 2, that for the adhesive layer with the granulated powdered terpolymer, and the extruder for the in-between adhesion promoter layer 20 with a mixture of 50 parts by weight of recycled protective film from Example 1 and 50 parts by weight of granulated adhesive from this example.

The bond strength of the resulting protective film on steel is 0.2 N/cm and 25 on paint 0.2 N/cm (fresh) and 1.4 N/cm (following storage under alternating climatic conditions). The protective film was bonded to freshly painted metal panels (2-component PU paint) and peeled off after a week; no paint film deformation was in evidence.

30 Bonded metal panels of the same type were subjected to UV ageing (1750 h of Xenotest 150, corresponding to 97 kLy). Following the removal of the protective film no residues are in evidence, not even in the edge region. Samples bonded to paint were weathered at 97 kLy; following removal, no residues could be seen on the surface or in the edge region.

35

Example 4

The protective film was produced as in Example 1 but with the following amendments: the adhesive composition consisted of 30% by weight of a terpolymer (56% by weight ethylene, 38.8% by weight propene and 5.2% by weight dicyclopentadiene, having a Mooney viscosity ML (1+4) 125°C of 43), 55% by weight of a terpolymer prepared with metallocene catalyst (51% by weight ethylene, 44% by weight propene and 5% by weight 5-ethylidene-2-norbornene, with Mooney viscosity ML (1+4) 125°C of 20), and with addition of 0.1% by weight each of Irganox 1070 and Chimassorb 944, 5% by weight of a terpene resin (Resin PC 1150, Yasuhara Chemical), 15% by weight of a liquid polybutene (Hyvis 5, BP Chemicals) and 2.5% by weight of triallyl cyanurate. The adhesive composition was crosslinked under nitrogen with electron beams (dose: 10 kGy).

15 The bond strength on steel is 0.8 N/cm, on paint 0.9 N/cm (fresh) and 2.2 N/cm (following storage under alternating climatic conditions). The protective film was bonded to freshly painted metal panels (2-component PU paint) and peeled off after a week; no paint film deformations are in evidence in the edge region. Samples bonded to paint were weathered at 97 kLy; following removal, no residues could be seen on the surface or in the edge region.

20

Comparative Examples

25 Comparative Example 1

Preparation is as in Example 1. The pressure-sensitive adhesive employed is a copolymer consisting of 80 mol% ethylene and 20 mol% 1-butene. The solvent used was hot toluene. The bond strength on paint is 0.2 N/cm (fresh) and 4.9 N/cm (following storage under alternating climatic conditions). The protective film tears when peeled off from the painted metal panel when pulling on one corner of an extensive bond. A protective film in accordance with this example exhibits severe paint film deformation.

35

Comparative Example 2

As Comparative Example 1; the copolymer consists of 90 mol% ethylene and 10 mol% 1-butene. Hot toluene is necessary as the solvent. The bond strength on painted sheet metal and on steel is less than 0.1 N/cm.

5

Comparative Example 3

As Example 1 but with a terpolymer of 80 mol% ethylene and 10 mol% each of propene and 1-butene. The bond strength on paint is 0.5 N/cm (fresh) and 4.2 N/cm (following storage under alternating climatic conditions). The protective film according to this example exhibits severe paint film deformation.

10

Comparative Example 4

As Example 1, but with a copolymer of 70 mol% vinyl acetate and 30 mol% ethylene. The bond strength on steel is 1.0 N/cm, on paint 0.9 N/cm (fresh) and 3.6 N/cm (following storage under alternating climatic conditions).

15

When bonded to fresh paint, severe deformations can be observed.

Comparative Example 5

The preparation is as in Example 1, with the adhesive employed being a mixture of polyisobutylenes; 25 parts by weight have a molecular weight M_w of 1.2 million, 75 parts by weight an M_w of 35 thousand. The bond strength on paint is 0.6 N/cm (fresh) and 1.8 N/cm (following storage under alternating climatic conditions). Samples bonded to paint were weathered at 97 kLy; following their removal, only slight residues were to be seen on the surface but very severe residues in the edge region. When bonded to fresh paint, slight paint film deformations are in evidence in the edge region.

25

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Patent Claims

1. Self-adhesive protective film for the exterior of painted vehicles, especially cars, characterized in that
 - 5 - the backing of the protective film is a film coated on one side with an adhesive composition
 - whose bond strength on steel is between 0.2 and 2.8 N/cm,
 - the adhesive composition comprising a copolymer of at least two different α -olefins having 2 to 12 carbon atoms and at least one diene as further comonomer, no α -olefin having a proportion of 75 mol% or more in the adhesive composition as a whole, and the copolymer having a Mooney viscosity ML (1+4) 125°C of less than 50.
- 15 2. Self-adhesive protective film according to Claim 1, characterized in that the proportion of diene in the adhesive composition is between 0.5 and 10% by weight.
- 20 3. Self-adhesive protective film according to Claim 1, characterized in that the UV permeability in the range from 290 to 360 nm is less than 1%.
- 25 4. Self-adhesive protective film according to Claim 1, characterized in that the copolymer of the adhesive composition has a Mooney viscosity ML (1+4) 125°C of less than 30.
5. Self-adhesive protective film according to Claim 1, characterized in that the adhesive composition is crosslinked.
- 30 6. Self-adhesive protective film according to Claim 1, characterized in that the proportion of polar comonomers in the copolymer is less than 20 mol%.
- 35 7. Self-adhesive protective film according to Claim 1, characterized in that it comprises at least one light stabilizer.
8. Self-adhesive protective film according to Claim 1, characterized in that it comprises at least one light stabilizer of the HALS class.

9. Self-adhesive protective film according to Claim 1, characterized in that no more than 65 mol% of any α -olefin is present in the copolymer.

5

10. Self-adhesive protective film according to Claim 1, characterized in that the bond strength on steel is between 0.3 and 1.5 N/cm.

10

11. Self-adhesive protective film according to Claim 1, characterized in that the proportion of each α -olefinic monomer in the copolymer is between 5 and 60 mol%.

15

12. Self-adhesive protective film according to Claim 1, characterized in that the UV permeability in the range from 290 to 360 nm is less than 0.1% and the backing film comprises one or more light stabilizers in an amount of at least 0.15% by weight.

20

13. Self-adhesive protective film according to Claim 1, characterized in that there is an adhesion promoter between the backing film and the adhesive composition.

25

14. Self-adhesive protective film according to Claim 1, characterized in that the force at 10% extension does not exceed 25 N/15 mm width either in the lengthwise or in the transverse direction.

15. Self-adhesive protective film according to Claim 1, characterized in that the adhesion promoter comprises at least one polymer of which at least 50 mol% consists of one or more α -olefins.

30

16. Self-adhesive protective film according to Claim 1, characterized in that the backing film comprises at least one copolymer of propylene.

35

17. Self-adhesive protective film according to Claim 1, characterized in that the adhesive composition and the backing film are joined to the protective film by simultaneous coextrusion.

18. Use of a protective film according to one of Claims 1 to 17 on curved surfaces on the exterior of cars.

19. Use of a protective film according to Claim 18, characterized in that the protective film is applied to painted surfaces before the vehicle is assembled.

Abstract

Self-adhesive protective film for the exterior of painted vehicles, especially cars, characterized in that

- the backing of the protective film is a film coated on one side with an adhesive composition
- whose bond strength on steel is between 0.2 and 2.8 N/cm,
- the adhesive composition comprising a copolymer of at least two different α -olefins having 2 to 12 carbon atoms and at least one diene as further comonomer, no α -olefin having a proportion of 75 mol% or more in the adhesive composition as a whole, and the copolymer having a Mooney viscosity ML (1+4) 125°C of less than 50.

COMBINATION DECLARATION & POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled „**Self-adhesive protective film with olefin rubber adhesive**“ the specification of which is attached hereto.

-OR-

was filed on _____ as

Application Serial No. _____ and was amended _____

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s)			<u>Priority Claimed</u>
<u>197 42 805.3</u> (Number)	<u>Germany</u> (Country)	<u>27 September 1997</u> (Day/Month/Yr. Filed)	<input checked="" type="checkbox"/> yes <input type="checkbox"/> no
_____ (Number)	_____ (Country)	_____ (Day/Month/Yr. Filed)	<input checked="" type="checkbox"/> yes <input type="checkbox"/> no

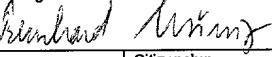
I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

<u>(Application Serial No.)</u>	<u>(Filing Date)</u>	<u>(Status)</u> (patented, pending, abandoned)
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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punished by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

POWER OF ATTORNEY: As a named Inventor, I hereby appoint the following attorneys and/or agents to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:
 Arnold Sprung, Reg. No. 17,232; Nathaniel D. Kramer, Reg. No. 25,350; Ira J. Schaefer, Reg. No. 26,802, and Esther Steinhauer, Reg. No. 40,255 all of 120 White Plains Road, Tarrytown, New York 10591; Kurt G. Briscoe, Reg. No. 33,141; William C. Gerstenzang, Reg. No. 27,552; Paul J. Juettner, Reg. No. 20,974 of 660 White Plains Road, New York 10591, my attorneys with full power of substitution and revocation

Send Correspondence To: Sprung Kramer Schaefer & Briscoe 660 White Plains Road 4th Fl. Tarrytown, New York 10591-5144	Direct Telephone Calls To: (914) 332-1700
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Full Name Of Sole or First Inventor MÜSSIG, Bernhard	Inventor's Signature 	Date 1,9.93
Residence Eddelsener Weg 31, D-21218 Seevetal, Germany		Citizenship German
Post Office Address Eddelsener Weg 31, D-21218 Seevetal, Germany		
Full Name Of Second Inventor	Inventor's Signature	Date
Residence		Citizenship
Post Office Address		
Full Name Of Third Inventor	Inventor's Signature	Date
Residence		Citizenship
Post Office Address		
Full Name Of Fourth Inventor	Inventor's Signature	Date
Residence		Citizenship
Post Office Address		
Full Name Of Fifth Inventor	Inventor's Signature	Date
Residence		Citizenship
Post Office Address		
Full Name Of Sixth Inventor	Inventor's Signature	Date
Residence		Citizenship
Post Office Address		
Full Name Of Seventh Inventor	Inventor's Signature	Date
Residence		Citizenship
Post Office Address		